bonds and using its lone pair electrons to accept a hydrogen bond from the neighbouring hydrazinium ion. The N(1) atom thus takes part in three hydrogen bonds, twice as the donor and once as the acceptor of the hydrogen atom. The hydrogen atom H(1) is bonded to oxygen of the sulphate ion and the N(1)–H(1)···O(4) hydrogen bond is bent with the N(1)–H(1) direction making an angle 44° whereas Cuthbert & Petch (1963) report 39°. The other hydrogen atom H(2) is bonded to nitrogen of the neighbouring NH_2 group and the N(1)–H(2) direction makes an angle of 18° with the N(1)–N(1) direction, which is more than the value of 4.5° reported by Cuthbert & Petch (1963). The hydrogen atoms of the NH_3 group are bonded to the nearest neighbouring acceptor oxygen atoms of the sulphate ion. The bonds are not collinear but bent, the angles being 123–152°.

We thank Mr G. Venkataraman for help in taking the inelastic spectrum.

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The Crystal Structure of β-DL-Arabinose

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The crystal structure of β-DL-arabinose ($C_5H_{10}O_5$) has been determined from integrated photographic intensity data by the application of a sign-correlation procedure on an IBM 1620 computer. The structure was refined by anisotropic least squares to give a final R value of 0·076 for 1122 reflections. The space group is $P2_1/c$, with $Z=4$ and unit-cell dimensions $a=5{\cdot}925$ ($\sigma=0{\cdot}004$), $b=7{\cdot}820$ ($\sigma=0{\cdot}003$), $c=13{\cdot}354$ ($\sigma=0{\cdot}006$) Å and $\beta=99{\cdot}45°$ ($\sigma=0{\cdot}06$). All the hydrogen atoms were located on difference syntheses, but were not refined. It is found that the bond C(1)–O(1)H is 1·392 Å and is significantly shorter than the mean value of 1·423 Å for the other C–OH bonds. There is no significant difference between the two ring C–O distances. These observations are consistent with those from other recent structure determinations of pyranose sugars. The D and L molecules are hydrogen-bonded in pairs across the centers of symmetry and throughout the crystal structure by centrosymmetrically related chains of hydrogen bonds, each of which links molecules of the same sense. All the oxygen atoms in the structure are involved in this hydrogen-bonding scheme.

Introduction

Both D- and L-arabinose are of biological origin. L-Arabinose occurs naturally as a free sugar and is widely distributed in the combined state in plant products, usually as a furanoside. D-Arabinose is found rarely but does occur in certain plants and in the polysaccharide of tubercle bacilli. The DL material has the trivial name 'pectinose', presumably because it can be obtained from the degradation of the polysaccharides in pectin substances.

When a DL mixture crystallizes, it may do so to form a racemic structure with a centric space group. This permits, at least in principle, a more straightforward solution to the phase problem and the attainment of a better accuracy by a factor of two in the atomic parameters (Cruickshank, 1950). We have taken advantage of this in DL-arabinose, and as far as we know, this is the first example of a centrosymmetric structure determination of a free DL sugar. The crystal structure of β-L-arabinose has been determined from a refinement of two projections by Hordvik (1961).

Crystal data

DL-Arabinose crystals were obtained by slow evaporation of an aqueous solution of the compound supplied by Mann Research Laboratories, Inc. The cell parameters were measured with Cu *Kα* radiation using a single-crystal orienter on a General Electric XRD-5 spectrogoniometer. The crystal density was measured at 23 °C by the flotation method in a mixture of carbon tetrachloride, chloroform and bromoform.

DL-Arabinose, $C_5H_{10}O_5$. M.W. 150·13.
Monoclinic, space group $P2_1/c$, from systematic absences: $h0l$ absent for $l=2n+1$, $0k0$ absent for $k=2n+1$.
$Z=4$.
$a=5{\cdot}925$ ($\sigma=0{\cdot}004$) Å.
$b=7{\cdot}820$ ($\sigma=0{\cdot}003$).
$c=13{\cdot}354$ ($\sigma=0{\cdot}006$). $\beta=99{\cdot}45$ ($\sigma=0{\cdot}06$)°.
$D_m=1{\cdot}638$ ($\sigma=0{\cdot}003$) g.cm^{-3}. $D_x=1{\cdot}634$ g.cm^{-3}.

This density is one per cent greater than that calculated from the X-ray data on the β-L form by Hordvik (1961).

Experimental

The integrated intensity data were recorded on multiple-film equi-inclination Weissenberg photographs using Cu *Kα* radiation. Using approximately cylindrical crystals of diameter 0·25 and 0·27 mm, four layers about the *a* axis and six layers about the *b* axis were taken. Of the 1520 theoretically accessible reflections unrelated by symmetry, 1122 reflections were observed above the background of the film, and their intensities were estimated visually by comparison with a standard scale. No correction for absorption or extinction was applied. These data were then correlated and reduced to the structure factors with an IBM 7090 program (Shiono, 1966) which uses the procedure described by Hamilton, Rollett & Sparks (1965).

Determination of the structure

The structure factors were converted to the normalized structure factors, $|E_{hkl}|$'s (Hauptman & Karle, 1953). The phase problem was then solved directly by a systematic sign correlation procedure using an IBM 1620 program prepared by Beurskens (1963). The signs of three linearly independent reflections were arbitrarily given positive signs in order to fix the origin and twelve more reflections with large $|E|$ values were given arbitrary signs. These 15 signs were then used to generate new signs by the Sayre (1952) relationship and their dependencies were traced back so that the most probable absolute signs could be determined, using the criterion based on the probability formula of Cochran & Woolfson (1955). This procedure gave 398 signs, which when used to compute a three-dimensional E map (Karle, Hauptman, Karle & Wing, 1958), clearly revealed the structure. The first structure factor calculation gave an R value of 0·30 for all the observed reflections.

Refinement of the structure

The refinement of the positional and thermal parameters was carried out by the Busing–Martin–Levy (1962) full-matrix least-squares IBM 7090 program.

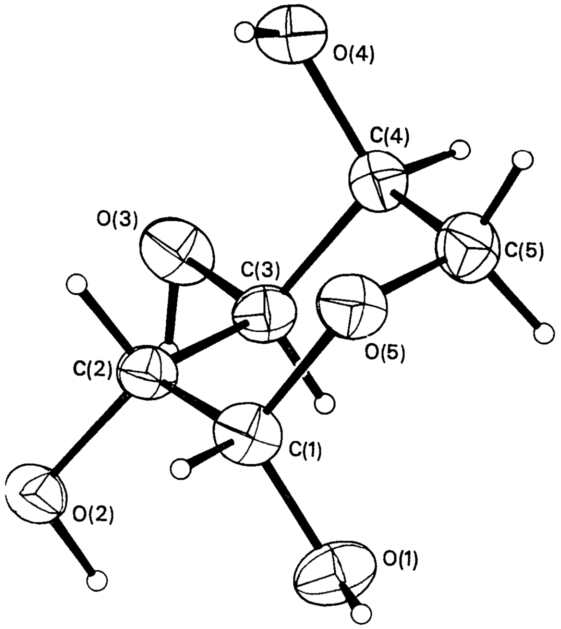

Fig. 1. Identification and numbering of the atoms in the molecule.

Table 1. *Fractional atomic coordinates* in β-DL-arabinose*

	x	y	z		x	y	z
C(1)	−0·0625 (5)	0·0680 (4)	0·6285 (2)	H(C1)†	0·105	0·033	0·646
C(2)	−0·1700 (5)	0·1350 (4)	0·7174 (2)	H(C2)	−0·070	0·228	0·747
C(3)	−0·4148 (5)	0·1937 (4)	0·6779 (2)	H(C3)	−0·486	0·099	0·650
C(4)	−0·4143 (5)	0·3302 (4)	0·5957 (2)	H(C4)	−0·570	0·362	0·566
C(5)	−0·2954 (6)	0·2589 (5)	0·5128 (2)	H(C5–1)	−0·380	0·168	0·477
O(1)	−0·1809 (4)	−0·0784 (3)	0·5905 (2)	H(C5–2)	−0·280	0·350	0·470
O(2)	−0·1600 (4)	0·0112 (3)	0·7956 (2)	H(O1)	−0·079	−0·120	0·555
O(3)	−0·5225 (4)	0·2610 (3)	0·7577 (2)	H(O2)	−0·280	−0·080	0·776
O(4)	−0·3139 (4)	0·4869 (3)	0·6356 (2)	H(O3)	−0·544	0·157	0·770
O(5)	−0·0668 (4)	0·2000 (3)	0·5536 (2)	H(O4)	−0·170	0·490	0·643

* The estimated standard deviations are given in parentheses and refer to the last decimal positions of respective values.
† The symbol in the parenthesis is the atom to which the hydrogen atom is bonded.

The Cruickshank (1961) weighting scheme was used and the variables included the 90 positional and thermal parameters for the carbon and oxygen atoms and one scale factor. Two cycles of isotropic refinement and two cycles of anisotropic refinement reduced R to 0·11. The difference Fourier synthesis was then examined and all the hydrogen atoms were revealed at reasonable locations with respect to the carbon and oxygen atoms. Inclusion of the hydrogen atoms and three more cycles of anisotropic refinement gave $R=0{\cdot}076$, at which stage the refinement was terminated to give positional and thermal parameters listed in Tables 1, 2 and 3. The corresponding structure factors on the absolute scale are given in Table 4. The scattering factors of Berghuis, Potters, Loopstra, MacGillavry & Veenendaal (1955) were used. The positional parameters of the hydrogen

Table 2. *Anisotropic thermal parameters* in β-*DL*-arabinose*

The temperature factor expression used was $\exp\{-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+2hk\beta_{12}+2hl\beta_{13}+2kl\beta_{23})\}$.

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	0·0159 (9)	0·0093 (5)	0·0034 (2)	0·0010 (6)	0·0026 (3)	0·0002 (2)
C(2)	0·0148 (8)	0·0066 (5)	0·0028 (2)	0·0000 (5)	0·0013 (3)	0·0003 (2)
C(3)	0·0143 (8)	0·0075 (5)	0·0031 (2)	0·0012 (5)	0·0019 (3)	−0·0004 (2)
C(4)	0·0165 (8)	0·0089 (5)	0·0027 (2)	0·0004 (6)	0·0007 (3)	0·0002 (2)
C(5)	0·0215 (10)	0·0109 (6)	0·0028 (2)	0·0014 (6)	0·0021 (3)	0·0001 (2)
O(1)	0·0216 (7)	0·0092 (4)	0·0046 (1)	−0·0013 (4)	0·0047 (3)	−0·0015 (2)
O(2)	0·0158 (6)	0·0078 (4)	0·0030 (1)	0·0001 (4)	0·0012 (2)	0·0009 (2)
O(3)	0·0197 (7)	0·0100 (4)	0·0035 (1)	0·0044 (4)	0·0041 (2)	0·0007 (2)
O(4)	0·0171 (7)	0·0076 (4)	0·0040 (1)	−0·0001 (4)	0·0018 (2)	0·0000 (2)
O(5)	0·0186 (7)	0·0094 (4)	0·0033 (1)	0·0006 (4)	0·0034 (2)	0·0003 (2)

* The estimated standard deviations are given in parentheses, and refer to the last decimal positions of respective values.

Table 3. *Principal axes of thermal ellipsoids*

The root mean square displacement U_i corresponds to the ith principal axis of the ellipsoid and $\theta_{ia},\theta_{ib},\theta_{ic}$ are angles between the ith axis and the crystallographic axes a,b,c.

	i	B_i*	U_i	θ_{ia}	θ_{ib}	θ_{ic}
C(1)	1	1·720 Å^2	0·1476 Å	146·2°	79·8°	48·7°
	2	2·215	0·1675	86·0	155·0	66·5
	3	2·694	0·1847	56·5	67·5	50·4
C(2)	1	1·570	0·1410	83·9	19·1	108·9
	2	1·919	0·1559	50·4	107·7	143·7
	3	2·062	0·1616	40·2	83·0	60·3
C(3)	1	1·478	0·1368	131·0	50·5	59·2
	2	2·088	0·1626	126·8	140·0	70·8
	3	2·279	0·1699	62·7	95·3	37·5
C(4)	1	1·827	0·1521	76·6	105·0	27·8
	2	2·186	0·1664	80·7	160·1	108·8
	3	2·328	0·1717	16·4	77·2	109·6
C(5)	1	1·815	0·1516	108·1	86·3	9·2
	2	2·519	0·1786	62·8	149·8	82·6
	3	3·089	0·1978	33·6	60·1	84·6
O(1)	1	1·755	0·1491	122·9	53·9	47·1
	2	2·247	0·1687	130·3	138·4	75·1
	3	4·051	0·2265	57·6	107·8	46·7
O(2)	1	1·621	0·1433	94·2	140·5	50·6
	2	2·155	0·1652	173·8	89·6	86·5
	3	2·349	0·1725	94·6	50·5	39·6
O(3)	1	1·372	0·1318	138·7	66·7	49·6
	2	2·155	0·1652	93·1	40·4	128·9
	3	3·718	0·2170	48·8	59·1	64·5
O(4)	1	1·863	0·1536	86·9	3·6	88·6
	2	2·279	0·1699	170·0	87·3	70·7
	3	2·817	0·1889	80·5	92·4	19·4
O(5)	1	1·615	0·1430	133·2	94·0	33·8
	2	2·274	0·1697	78·1	166·7	86·3
	3	2·993	0·1947	45·7	77·4	56·5

* $B_i=8\pi^2U_i^2$.

Table 4. *Observed and calculated structure factors*

Columns are: Index, $|F_{\text{obs}}|$, $F_{\text{cal.}}$ (* unobserved).

K= 0 H= 0		
2	329	292-
4	21	5-
6	254	251-
8	90	92
10	42	42
12	47	40-
14	32	34
16	20	16
K= 1 H= 0		
1	134	138-
2	512	532-
3	197	180-
4	85	69-
5	217	203
6	173	156-
7	63	73
8	123	113-
9	52	49
10	10*	6
11	30	28-
12	34	37-
13	33	29-
14	31	27
15	59	50
16	8*	3-
K= 2 H= 0		
0	100	108-
1	187	207
2	205	208-
3	297	316-
4	618	624-
5	61	38-
6	157	135
7	265	232
8	24	16
9	72	79
10	116	113
11	24	18
12	165	178-
13	131	134-
14	106	117
15	46	44-
16	24	26-
K= 3 H= 0		
1	250	255
2	104	107
3	343	349-
4	304	298
5	341	327
6	162	151
7	119	108-
8	27	20
9	37	42-
10	149	156-
11	139	145-
12	117	120
13	32	31-
14	36	35
15	64	69
16	14	17
K= 4 H= 0		
0	366	382
1	84	88-
2	171	151-
3	123	130-
4	145	138
5	111	104-
6	94	100
7	137	133-
8	112	100
9	79	67
10	82	75-
11	69	80
12	10*	5-
13	14	9
14	18	13
15	20	26
K= 5 H= 0		
1	137	134
2	57	60
3	215	212-
4	22	23-
5	89	84-
6	132	128
7	31	26
8	210	211-
9	195	205
10	67	61-
11	9*	0
12	10*	1-
13	16	8
14	46	46
K= 6 H= 0		
0	49	50
1	9*	1-
2	66	76-
3	80	88
4	83	81-
5	10*	13-
6	76	76
7	116	116
8	124	124-
9	33	42-
10	37	36
11	8*	16-
12	90	88-
13	6*	9-
K= 7 H= 0		
1	206	201
2	129	130-
3	132	133-
4	56	48-
5	95	88-
6	64	64
7	20	18-
8	9*	10
9	17	16-
10	42	34
11	7*	11-
12	13	13
K= 8 H= 0		
0	27	29
1	23	24
2	121	118-
3	98	95-
4	134	134
5	59	57
6	25	22
7	17	14-
8	98	80
9	31	26-
10	5*	9
K= 9 H= 0		
1	36	29
2	9*	9
3	59	43-
4	29	23-
5	29	26
6	47	41
7	51	44
K= 10 H= 0		
0	35	35
K= 0 H= 1		
0	18	12-
2	975	987
4	179	145
6	216	197-
8	196	172-
10	75	71
12	192	194-
14	172	161
16	8*	4
-2	553	478-
-4	430	413-
-6	198	177
-8	235	222-
-10	48	40-
-12	190	201-
-14	33	36
-16	10*	8-
K= 1 H= 1		
0	460	531
1	587	680-
2	170	182
3	198	174
4	90	70
5	42	44
6	32	22-
7	110	91-
8	22	17-
9	183	163
10	82	74-
11	22	12-
12	126	125
13	12*	1
14	80	73
15	29	25-
16	20	22
-1	65	54-
-2	64	49
-3	112	101-
-4	282	264
-5	658	632
-6	367	354
-7	34	25-
-8	10*	8
-9	277	285-
-10	163	170
-11	146	145
-12	178	200-
-13	21	24-
-14	38	36-
-15	25	21
-16	25	28-
-17	17	27-
K= 2 H= 1		
0	438	486
1	288	303
2	305	295-
3	183	175
4	243	230
5	115	115-
6	405	432-
7	46	43-
8	150	144
9	76	68
10	102	99
11	128	127
12	11*	0
13	51	54-
14	65	57-
15	11*	10-
16	6*	0
-1	51	48-
-2	461	493-
-3	81	69-
-4	27	12-
-5	386	377-
-6	355	344
-7	98	86
-8	78	78
-9	41	30-
-10	95	93
-11	80	81
-12	149	164
-13	70	81
-14	38	40-
-15	26	23-
-16	17	13
K= 3 H= 1		
0	179	190-
1	241	255
2	250	256-
3	209	203
4	140	125
5	103	90-
6	156	141
7	215	196-
8	37	25-
9	166	156
10	91	84-
11	63	65-
12	126	132-
13	51	54-
14	34	36
15	16	19-
-1	51	51
-2	301	333-
-3	28	33
-4	162	169-
-5	100	96
-6	73	82-
-7	269	260-
-8	109	108
-9	116	112-
-10	34	34-
-11	47	47
-12	45	49-
-13	35	42
-14	12*	0
-15	24	22
-16	29	39
K= 4 H= 1		
0	118	124-
1	150	156
2	125	120
3	238	226-
4	46	34
5	248	253-
6	109	102-
7	30	31-
8	70	63
9	12*	7-
10	86	83
11	50	44
12	47	47-
13	23	18-
14	21	19
-1	147	151
-2	122	122
-3	62	66
-4	196	191-
-5	267	271
-6	198	214
-7	150	152-
-8	340	381-
-9	76	84-
-10	38	39
-11	31	30-
-12	36	34-
-13	22	20
-14	11*	2
-15	19	22
K= 5 H= 1		
0	23	21-
1	11*	4-
2	9*	1-
3	19	13
4	25	17-
5	39	40
6	209	208
7	160	158-
8	72	68
9	119	121
10	29	23-
11	43	42
12	21	19-
13	10*	8
14	6*	8
-1	95	94-
-2	101	114
-3	284	326-
-4	24	26
-5	187	192
-6	83	84
-7	116	128
-8	52	46
-9	12*	2-
-10	12*	10-
-11	48	41
-12	32	32-
-13	27	22-
-14	20	19
K= 6 H= 1		
0	26	22-
1	83	79
2	105	99-
3	13*	17
4	187	188
5	113	105
6	98	92-
7	49	48-
8	40	36
9	135	128
10	49	46-
11	33	27-
12	60	51-
-1	23	25-
-2	91	93-
-3	132	128-
-4	237	257
-5	23	28-
-6	53	60
-7	14*	1
-8	53	61-
-9	13*	17
-10	47	50-
-11	11*	5-
-12	47	49
-13	65	71
K= 7 H= 1		
0	100	100
1	90	79
2	14*	6
3	198	200
4	165	157-
5	98	95-
6	35	31-
7	61	59-
8	76	73-
9	23	15
10	19	14-
11	72	68-
-1	39	37
-2	93	90-
-3	135	137-
-4	13*	5-
-5	23	23
-6	27	32-
-7	41	38
-8	24	26
-9	44	43-
-10	12*	5
-11	32	36
-12	31	32-
K= 8 H= 1		
0	53	47-
1	41	38
2	89	86-
3	28	22-
4	21	16-
5	41	45-
6	33	22
7	12*	5
8	43	34-
9	80	70-
-1	13*	11-
-2	13*	12-
-3	67	74-
-4	21	24
-5	46	47
-6	59	69
-7	32	25
-8	122	113-
-9	25	23
-10	14	14
K= 9 H= 1		
0	103	86
1	12*	10
2	12*	5-
3	12*	6
4	60	51
5	61	47-
6	15	15-
-1	34	33
-2	22	17-
-3	83	73-
-4	46	38-
-5	11*	16
-6	9*	1
-7	14	13-
K= 0 H= 2		
0	54	47
2	228	199-
4	434	424
6	130	135-
8	37	33-
10	141	135-
12	57	54-
14	129	114-
-2	64	75-
-4	422	349-
-6	281	251-
-8	31	23
-10	234	224-
-12	162	160
-14	89	94-
-16	121	114
K= 1 H= 2		
0	34	34-
1	575	632-
2	350	344-
3	52	36
4	8*	11
5	95	81-
6	59	50-
7	49	34-
8	37	29-
9	58	52
10	22	11-
11	13*	5-
12	113	114-
13	122	108
14	115	98
15	85	94-
-1	347	380
-2	88	81
-3	413	403
-4	416	394
-5	73	69-
-6	228	199-
-7	286	277-
-8	26	19
-9	39	35-
-10	297	315-
-11	236	238
-12	99	103-
-13	48	48-
-14	27	25-
-15	26	22-
-16	9*	0
K= 2 H= 2		
0	454	504-
1	21	15
2	192	210
3	179	169
4	94	89
5	40	30-
6	79	65
7	44	38
8	18	18
9	43	40-
10	11*	19
11	69	64
12	69	70
13	11*	11-
14	117	108
15	26	32-
-1	131	147-
-2	319	334
-3	203	211-
-4	121	103-
-5	94	74
-6	379	383
-7	9*	2-
-8	53	55
-9	80	78-
-10	168	172
-11	142	147
-12	108	115-
-13	10*	5
-14	64	79-
-15	19	15
-16	14	14
K= 3 H= 2		
0	26	26-
1	154	159-
2	171	184-
3	306	304
4	10*	9
5	220	224
6	213	201
7	84	86-
8	57	59
9	105	104-
10	12*	1
11	12*	1
12	58	62-
13	25	27
14	18	20-
-1	111	118
-2	23	2
-3	91	83
-4	72	90
-5	195	199-
-6	145	138
-7	31	30
-8	34	28-
-9	136	135-
-10	118	129
-11	62	68
-12	12*	14-
-13	22	27-
-14	89	93
-15	46	45-
-16	33	39
K= 4 H= 2		
0	9*	16-
1	218	232
2	302	312-
3	161	164
4	262	279
5	136	125-
6	217	232-
7	273	305-
8	36	38
9	45	42
10	129	141-
11	34	39
12	42	42
13	30	25
14	22	28-
-1	39	46
-2	62	60
-3	45	47
-4	27	25-
-5	43	37-
-6	114	111-
-7	11*	2
-8	66	77-
-9	12*	6
-10	34	33
-11	12*	7
-12	30	38
-13	12*	6
-14	57	58-
-15	53	65-
K= 5 H= 2		
0	9*	0
1	195	201-
2	62	59-
3	26	29
4	18	25-
5	101	98-
6	12*	11-
7	31	28
8	39	37
9	11*	9-
10	11*	13
11	12*	1-
12	20	20
13	46	54
-1	106	108-
-2	29	22-
-3	83	91
-4	49	46-
-5	28	17
-6	37	33-
-7	88	95
-8	44	45-
-9	40	40
-10	32	29-
-11	49	50
-12	20	18-
-13	90	87-
-14	88	98-
K= 6 H= 2		
0	51	48-
1	13*	10
2	26	29-
3	13*	0
4	32	25
5	110	97
6	70	69
7	12*	17-
8	12*	6
9	36	31
10	98	76
11	53	45
12	16	19
-1	116	112-
-2	342	375
-3	162	179-
-4	54	50-
-5	105	111
-6	62	65-
-7	99	110-
-8	148	167-
-9	12*	20
-10	61	72
-11	54	48
-12	99	100
-13	41	46
K= 7 H= 2		
0	35	31-
1	68	69-
2	29	25
3	134	135
4	36	39
5	75	70
6	51	53-
7	41	33-
8	12*	1-
9	40	30
10	8*	2-
-1	104	108
-2	93	96
-3	102	104-
-4	47	44
-5	12*	3
-6	86	93
-7	12*	14
-8	23	32-
-9	23	26-
-10	11*	13
-11	9*	6
-12	15	18-
K= 8 H= 2		
0	100	104-
1	47	43-
2	73	70-
3	13*	7-
4	13*	6-
5	35	30
6	60	48-
7	10*	18-
8	8*	11-
-1	12*	8-
-2	31	33
-3	79	87
-4	73	77
-5	86	75
-6	90	84-
-7	12*	16
-8	21	23
-9	74	76-
-10	17	22
K= 9 H= 2		
0	56	50
1	70	64-
2	10*	2-
3	53	47
4	37	33
5	7*	5-
-1	19	17-
-2	188	170-
-3	44	39-
-4	10*	8
-5	9*	0
-6	32	32-
K= 0 H= 3		
0	248	253-
2	554	593
4	137	136-
6	252	248
8	77	73
10	136	133-
12	12*	2-
14	26	25-
-2	219	169
-4	365	311-
-6	278	245
-8	122	107
-10	73	73-
-12	33	22-
-14	72	70
-16	170	168
K= 1 H= 3		
0	222	234
1	57	31
2	41	36-
3	70	63-
4	239	247-
5	105	92-
6	44	37-
7	21	17
8	124	129-
9	39	38-
10	13*	3-
11	22	23-
12	28	25-
13	103	96
14	50	53-
-1	240	251
-2	195	198
-3	21	1-
-4	90	87-
-5	327	308-
-6	36	32-
-7	142	128
-8	173	181-
-9	125	123
-10	13*	7
-11	13*	2-
-12	13*	16-
-13	12*	5-
-14	13*	3-
-15	84	84-
-16	8*	2-
K= 2 H= 3		
0	52	45-
1	153	153-
2	290	307-
3	175	190
4	90	93-
5	21	25-
6	108	107-
7	70	67-
8	158	165
9	51	52
10	60	68-
11	12*	3
12	19	22
13	61	56
14	7*	14-
-1	100	98-
-2	45	31
-3	165	170-
-4	48	41-
-5	77	61-
-6	26	27
-7	48	42
-8	196	195
-9	112	102
-10	263	287-
-11	88	97
-12	19	17-
-13	30	33-
-14	52	50-
-15	11*	5-
-16	14	17-
K= 3 H= 3		
0	54	59
1	37	36
2	107	121-
3	29	29
4	43	38-
5	106	110-
6	91	95
7	248	269
8	133	140
9	74	81-
10	66	70
11	57	55-
12	22	17-
13	9*	1-
-1	54	51
-2	79	84
-3	75	77
-4	84	78
-5	265	292-
-6	158	167-
-7	12*	4
-8	111	114
-9	93	94-
-10	32	35-
-11	61	50
-12	58	60
-13	13*	15
-14	19	16-
-15	58	64
K= 4 H= 3		
0	60	58
1	11*	3
2	97	93
3	122	127
4	102	101-
5	134	135
6	91	88
7	76	79-
8	35	41
9	113	132-
10	26	24-
11	12*	9
12	35	34-
13	6*	8
-1	32	28
-2	191	205
-3	35	34-
-4	137	143-
-5	152	170
-6	25	19
-7	12*	8
-8	62	62
-8	64	62
-9	91	92-
-9	86	92-
-10	52	59
-11	33	30-
-12	22	17
-13	55	56-
-14	67	68
-15	27	39-
K= 5 H= 3		
0	51	56-

Table 4 (*cont.*)

1	104	114-
2	125	134-
3	12•	2
4	12•	10-
5	85	86-
6	41	41-
7	42	45
8	34	36
9	10•	12-
10	90	80-
11	9•	8-
12	24	30-
-1	45	43
-2	119	132
-3	34	32
-4	85	88-
-5	12•	13-
-6	61	65
-7	247	288
-8	87	92-
-9	29	28-
-10	53	54
-11	94	90-
-12	89	76-
-13	9•	6
-14	49	62
K= 6 H= 3		
0	53	42
1	62	57-
2	41	42
3	13•	9-
4	13•	26-
5	50	54
6	40	35-
7	29	25-
8	30	25
9	40	32
10	103	105-
-1	135	146-
-2	13•	8-
-3	53	55
-4	141	151-
-5	89	93-
-6	139	152-
-7	32	32
-8	71	78
-9	51	55
-10	11•	10-
-11	46	43
-12	10•	2
K= 7 H= 3		
0	130	130
1	48	46
2	13•	18-
3	12•	13
4	54	53
5	11•	6-
6	26	19-
7	80	66
8	10•	9-
9	7•	2
-1	50	51-
-2	50	47
-3	65	66-
-4	22	20-
-5	22	21-
-6	12•	9-
-7	35	36
-8	10•	21-
-9	29	31-
-10	10•	9
-11	69	75-
K= 8 H= 3		
0	51	42-
1	44	42-
2	13•	7
3	12•	2
4	80	67-
5	25	23-
6	28	27
7	5•	7
-1	174	156
-2	218	205
-3	13•	0
-4	87	81-
-5	12•	0
-6	38	40
-7	65	57-
-8	51	52
-9	6•	11-
K= 9 H= 3		
0	124	115-
1	8•	8-
2	7•	7
3	5•	30-
-1	36	29
-2	24	19-
-3	21	20
-4	22	22-
-5	6•	10
K= 0 H= 4		
0	125	95-
2	87	79
4	114	104
6	32	30-
8	225	229
10	23	18
12	9•	8-
-2	73	63-
-4	103	89
-6	52	44-
-8	86	80-
-10	254	259-
-12	144	147
-14	189	154
K= 1 H= 4		
0	143	142
1	175	173
2	189	199
3	11•	4-
4	38	33
5	40	43-
6	28	10-
7	33	34
8	21	13
9	66	60-
10	82	73-
11	26	22
12	9•	12-
-1	198	196-
-2	90	92
-3	87	79
-4	146	135
-5	11•	6
-6	138	120-
-7	46	36
-8	134	133
-9	58	61
-10	109	107-
-11	41	42-
-12	35	27
-13	32	26
-14	11•	10-
-15	84	87-
K= 2 H= 4		
0	115	120-
1	160	168-
2	197	215
3	53	45
4	253	294-
5	137	145
6	44	41-
7	20	20-
8	76	75-
9	9•	8-
10	37	35
11	20	19-
12	7•	3
-1	88	90-
-2	36	34-
-3	127	129-
-4	139	134
-5	211	222
-6	153	151
-7	70	71
-8	27	29-
-9	10•	3
-10	74	66-
-11	33	29-
-12	9•	10
-13	10•	2-
-14	35	32
-15	6•	8-
K= 3 H= 4		
0	11•	12-
1	63	58
2	12•	16-
3	147	173
4	171	182-
5	76	76-
6	12•	10-
7	12•	5
8	11•	23
9	13•	0
10	31	26
11	17	18
12	6•	5
-1	106	112-
-2	61	60
-3	97	91-
-4	270	290-
-5	151	156-
-6	12•	4
-7	61	58
-8	25	22-
-9	12•	0
-10	38	40
-11	11•	4
-12	13•	10-
-13	85	84
-14	9•	4-
-15	19	29-
K= 4 H= 4		
0	53	50
1	11•	16
2	134	145
3	62	64
4	21	17
5	29	31-
6	11•	7-
7	11•	1
8	13•	11
9	11•	12-
10	42	40
11	33	42-
-1	42	44-
-2	149	170-
-3	129	139
-4	93	92
-5	39	38-
-6	122	121-
-7	42	41-
-8	123	129
-9	77	81-
-10	94	104-
-11	41	39-
-12	46	44
-13	7•	15-
-14	13	16-
K= 5 H= 4		
0	177	219
1	51	56
2	38	38-
3	46	50-
4	11•	16-
5	101	124-
6	46	55-
7	86	77
8	83	84
9	9•	8-
10	14	18
-1	75	75-
-2	30	31
-3	111	121
-4	23	19-
-5	154	154
-6	41	37-
-7	22	22
-8	76	73
-9	107	119-
-10	29	21
-11	28	24-
-12	64	58
-13	11	13
K= 0 H= 5		
0	33	25-
2	34	37
4	62	59-
6	11•	9
8	124	95-
10	96	83
-2	144	129-
-4	54	62-
-6	151	142-
-8	192	194-
-10	168	162
-12	51	47
-14	40	40
K= 1 H= 5		
0	56	55
1	12•	2
2	87	87
3	42	37-
4	12•	0
5	67	70
6	12•	9
7	24	15-
8	22	16
9	92	79-
10	28	23
-1	90	91-
-2	153	150
-3	135	139
-4	221	207-
-5	57	49-
-6	33	30
-7	25	22-
-8	148	145-
-9	121	118
-10	25	22-
-11	69	56-
-12	21	17-
-13	60	50-
-14	16	15
K= 2 H= 5		
0	25	27
1	61	47-
2	10•	0
3	97	102-
4	68	61
5	24	18
6	30	28-
7	22	19
8	24	29-
9	57	50
10	29	29
-1	78	82-
-2	75	71
-3	159	164
-4	259	284
-5	76	80
-6	89	89-
-7	89	87-
-8	39	44
-9	22	16
-10	32	32-
-11	10•	1
-12	21	15
-13	56	51
-14	30	36
K= 3 H= 5		
0	12•	17
1	35	36-
2	58	52
3	35	36-
4	51	58-
5	114	141
6	61	73-
7	52	48
8	35	32
9	36	39-
-1	82	79-
-2	90	98-
-3	12•	1-
-4	25	24-
-5	76	73
-6	12•	8
-7	109	119-
-8	137	157
-9	45	50
-10	22	20
-11	55	48
-12	42	44
-13	8•	0
K= 4 H= 5		
0	133	148-
1	58	66-
2	28	24
3	69	84
4	53	55-
5	58	53
6	105	113
7	24	25-
8	94	114-
9	44	56-
-1	20	18
-2	32	33-
-3	27	25
-4	29	31
-5	26	26
-6	83	86-
-7	25	27-
-8	23	15-
-9	119	116-
-10	20	13-
-11	10•	18-
-12	8•	10-
K= 5 H= 5		
0	11•	4
1	10•	1
2	89	104
3	12•	1-
4	11•	4-
5	45	40-
6	9•	1-
7	77	87-
8	5•	12-
-1	19	20
-2	66	68-
-3	90	99
-4	53	51-
-5	10•	0
-6	20	19
-7	105	94-
-8	34	30
-9	52	45
-10	9•	2
-11	48	49-
K= 0 H= 6		
0	45	49
2	80	67-
4	125	84
6	11•	6-
8	7•	2
-2	166	157-
-4	49	37
-6	56	60-
-8	128	102
-10	112	92
-12	8•	11-
K= 1 H= 6		
0	38	38
1	21	16-
2	55	40
3	124	112-
4	38	35
5	68	62
6	55	45-
7	58	53
8	7•	7-
-1	71	71
-2	149	158-
-3	12•	3-
-4	35	33-
-5	12•	17
-6	92	75-
-7	37	24
-8	13•	0
-9	12•	7
-10	20	21-
-11	73	62-
-12	40	34-
K= 2 H= 6		
0	115	123
1	25	26-
2	9•	5-
3	47	33-
4	59	50-
5	19	15
6	32	31
7	32	35-
-1	9•	10-
-2	9•	9
-3	70	66
-4	44	48-
-5	36	35-
-6	9•	0
-7	11•	1-
-8	80	66-
-9	29	26
-10	15	12
-11	7•	5
-12	46	58-
K= 3 H= 6		
0	11•	21-
1	22	24
2	41	41
3	82	82-
4	23	19
5	31	33
6	9•	3
-1	36	41
-2	28	27
-3	57	58-
-4	49	48-
-5	47	51-
-6	164	147
-7	66	61-
-8	59	51
-9	165	154
-10	44	43
-11	33	35-
K= 4 H= 6		
0	48	48
1	12•	16
2	46	47-
3	18	24-
4	17	15-
5	17	25
6	6•	21
-1	21	15
-2	12•	4
-3	118	111
-4	12•	6-
-5	29	23-
-6	20	14-
-7	151	155-
-8	44	45
-9	26	28-
-10	33	40
K= 5 H= 6		
0	10•	7-
1	52	58
2	27	27-
3	57	71-
4	24	31-
-1	59	62
-2	65	62-
-3	42	43-
-4	36	32
-5	61	52
-6	64	65-
-7	18	17-
-8	57	61
K= 0 H= 7		
0	46	39
2	9•	11
4	19	23
-2	99	81-
-4	11•	18-
-6	102	90
-8	8•	5-
K= 1 H= 7		
0	22	23-
1	10•	10
2	10•	3-
3	18	16-
4	7•	41
-1	97	89
-2	35	35
-3	38	37-
-4	36	29-
-5	29	27
-6	49	45
-7	22	21-
-8	34	32-
-9	35	40-
K= 2 H= 7		
0	8•	5
1	8•	2
2	14	14
3	42	50-
-1	8•	0
-2	80	81-
-3	73	70
-4	24	21
-5	8•	15-
-6	35	33
-7	7•	10-
-8	49	50-
K= 3 H= 7		
0	30	32
1	8•	5
-1	28	31-
-2	91	94-
-3	87	98-
-4	56	55
-5	52	52
-6	28	28
-7	47	55
K= 4 H= 7		
-1	6•	51
-2	7•	6
-3	27	34-

Table 5. *Bond lengths and angles* in β*-DL-*arabinose and β*-L-*arabinose [in brackets]*

		d(i, j)					<(ijk)	
i	j	β-DL	[β-L]	i	j	k	β-DL	[β-L]
C(1)	C(2)	1·532 (4) Å	[1·524] Å	O(5)	C(1)	C(2)	109·5 (3)°	[109·4]°
C(2)	C(3)	1·528 (4)	[1·523]	O(5)	C(1)	O(1)	112·9 (3)	[113·1]
C(3)	C(4)	1·533 (4)	[1·554]	C(2)	C(1)	O(1)	108·3 (2)	[108·9]
C(4)	C(5)	1·515 (5)	[1·536]	C(1)	C(2)	C(3)	109·3 (2)	[109·2]
C(5)	O(5)	1·447 (4)	[1·440]	C(1)	C(2)	O(2)	111·5 (2)	[107·3]†
C(1)	O(5)	1·434 (4)	[1·421]	C(3)	C(2)	O(2)	112·2 (2)	[110·8]
C(1)	O(1)	1·392 (4)	[1·382]	C(2)	C(3)	C(4)	109·9 (2)	[109·7]
C(2)	O(2)	1·417 (4)	[1·454]†	C(2)	C(3)	O(3)	111·7 (2)	[109·2]†
C(3)	O(3)	1·432 (4)	[1·426]	C(4)	C(3)	O(3)	109·4 (2)	[110·1]
C(4)	O(4)	1·425 (4)	[1·431]	C(3)	C(4)	C(5)	108·9 (3)	[107·1]
				C(3)	C(4)	O(4)	112·5 (3)	[111·7]
				C(5)	C(4)	O(4)	111·9 (3)	[108·3]†
				C(4)	C(5)	O(5)	111·3 (3)	[111·9]
				C(5)	O(5)	C(1)	113·4 (3)	[112·7]

* The estimated standard deviations are given in parentheses, and refer to the last decimal positions of respective values.

† These results differ in the two studies by greater than 3σ.

atoms were not refined and they were assigned the same thermal parameters as those of the carbon or oxygen atoms to which they were bonded. The thermal ellipsoid parameters given in Table 3 and illustrated in Fig. 1 show that there is no marked thermal anisotropy. Neither is there any evidence of rigid-body motion which would increase the thermal displacements of the substituent atoms relative to those of the ring.

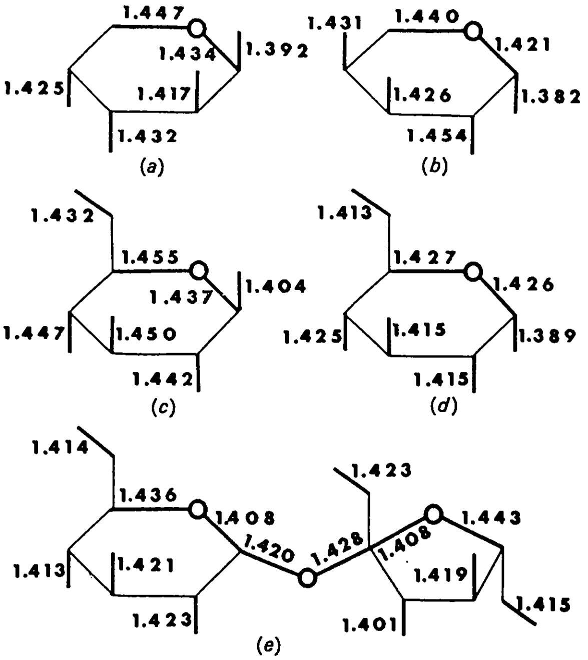

Fig. 2. C–O distance in (*a*) β-D,L-arabinose, (*b*) β-L-arabinose (*c*) β-D-glucose, (*d*) α-D-glucose, (*e*) sucrose.

Description of the structure

The conventional numbering and identification of the atoms for one molecule are shown in Fig. 1. The molecule is the β anomer with the chair-shaped pyranose ring. Its conformation is 1*a*2*e*3*e*4*a*, where *a* and *e* refer to the axial and equatorial positions for the hydroxyl groups.

The bond lengths and valence angles are given in Table 5. With the exceptions discussed below, they agree within the experimental errors with those obtained by the projection analysis of the β-L-arabinose by Hordvik (1961), also shown in Table 5. The C–C bond lengths range from 1·515 to 1·533 Å and none of these differ significantly from the mean value of 1·527 Å. The C–OH bond lengths, excluding C(1)–O(1)H, range from 1·417 to 1·428 Å with the mean value of 1·423 Å. The bond length of C(1)–O(1)H is 1·392 Å and is shorter than this mean value by 7σ. This relative shortening of the C(1)–O(1)H bond length has consistently been observed in previous structure determinations of pyranose sugars with comparable values, as shown below:

β-Arabinose: 1·392 *vs* 1·423 Å
$\Delta l = 7\sigma$ (this work).

β-Arabinose: 1·382 *vs* 1·437
$\Delta l = 4\sigma$ (Hordvik, 1961).

α-D-Glucose: 1·389 *vs* 1·418
$\Delta l = 10\sigma$ (Brown & Levy, 1965).

β-D-Glucose: 1·404 *vs* 1·446
$\Delta l = 4\sigma$ (Ferrier, 1963).

α-Rhamnose: 1·376 *vs* 1·435
$\Delta l = 2\sigma$ (McGeachin & Beevers, 1957)

Table 6

(*a*) Conformation angles in β-DL-arabinose and β-L-arabinose [in brackets].

Bond	Conformation angle*	
C(1) → C(2)	+58·2°	[+59·9°]
C(2) → C(3)	−57·0	[−58·4]
C(3) → C(4)	+55·4	[+55·4]
C(4) → C(5)	−55·8	[−55·9]
C(5) → O(5)	+59·6	[+60·9]
O(5) → C(1)	−60·0	[−61·3]

* The conformation angle of a directed bond C(2) → C(3) is defined as the angle, measured clockwise, that the projection of the bond C(3)–C(4) makes with respect to the projection of the bond C(1)–C(2).

(*b*) Range of the conformation angles in some pyranose rings and chair form carbon rings

*Ideal value	55·8–61·7°	
Sucrose	54·8–56·0	Brown & Levy (1963)
L-Sorbose	51·6–58·2	Kim & Rosenstein (1967)
β-DL-Arabinose	55·4–60·0	Kim & Jeffrey (this paper)
β-Arabinose	55·4–61·3	Hordvik (1961)
α-D-Glucose	51·3–62·2	Brown & Levy (1965)
β-D-Glucose	52·2–66·1	Ferrier (1963)
K, Rb-Glucuronate	49·1–68·3	Gurr (1963)
†Myoinositol	53·7–59·8	Rabinowitz & Kraut (1963)
†Myoinositol . $2H_2O$	56·0–60·1	Lomer, Miller & Beevers (1962)

* Hypothetical pyranose ring with C–C = 1·525 Å, C–O(ring) = 1·430 Å, valence angle on carbon atoms = 109·5° and that on ring oxygen atom = 113·3°.

† Chair form six-carbon ring.

where the differences, Δl, of bond lengths between average C–OH and C(1)–O(1)H are expressed as multiples of σ's, the estimated standard deviations of the differences.

This new result therefore adds strong support to the earlier observations and further establishes, as a general molecular characteristic of pyranose monosaccharides, that the C(1)–O(1)H link is about 0·04 Å shorter than the other C–OH. However, the fact that this shortening was not observed in sucrose (Brown & Levy, 1963) or in α-methyl-D-galactoside-6-bromohydrin (Robertson & Sheldrick, 1965) indicates that it is a property of the free hydroxyl group only. Instead, the glycosidic substituted molecules show differences in the ring C–O links, that adjacent to the glycosidic link being the shorter, a feature not observed in the presence of the O(1)H hydroxyl group. This interesting comparison between the C–O bond lengths is illustrated in Fig. 2. Unfortunately the results on cellobiose (Jacobson, Wunderlich & Lipscomb, 1961) are not sufficiently precise to be relevant. We have as yet only the one example of a mono-pyranoside substituted in the O(1) position quoted above, in which the accuracy is borderline (σ=0·016 Å) owing to the presence of the heavy atom, although the data were collected at 125°K.

The evidence for similar differences in the ring C–O bonds in furanosyl derivatives has recently been discussed by Sundaralingam (1965). There is also a trend for the observed ring C–O links to be longer than the C–OH by about 0·01 Å, but this has yet to be established at a good significance level. The carbon valence angles inside the ring range from 108·9 to 111·3° with the mean value of 109·8° and outside the ring range from 108·3 to 112·9° with the mean value of 111·3°. The valence angle of the ring oxygen is 113·4°, *i.e.* greater than the mean values within the ring, as has been commonly observed in other structures (*cf.* Jeffrey & Rosenstein, 1964). The conformation angles of the pyranose ring are given in Table 6 and their range is compared with that of an unstrained *trans* ring and with some other pyranose and cyclohexane rings. Clearly there is no evidence of strain in the ring caused by intermolecular packing or hydrogen bonding in the crystal.

In comparing the molecular dimensions with those reported for the β-L-arabinose, one C–O distance and three C–C–OH angles have differences exceeding 3σ, as indicated in Table 5. The C(2)–O(2) distance of 1·454 Å is larger than the mean, but it remains to be established whether there is any connection between this and the fact that O(2) is involved in three hydrogen bonds in the β-L structure, rather than the more usual one or two, as in the DL form. The three angles, all of which are smaller in the β-L form, could well be the consequence of the intermolecular hydrogen bond and packing forces.

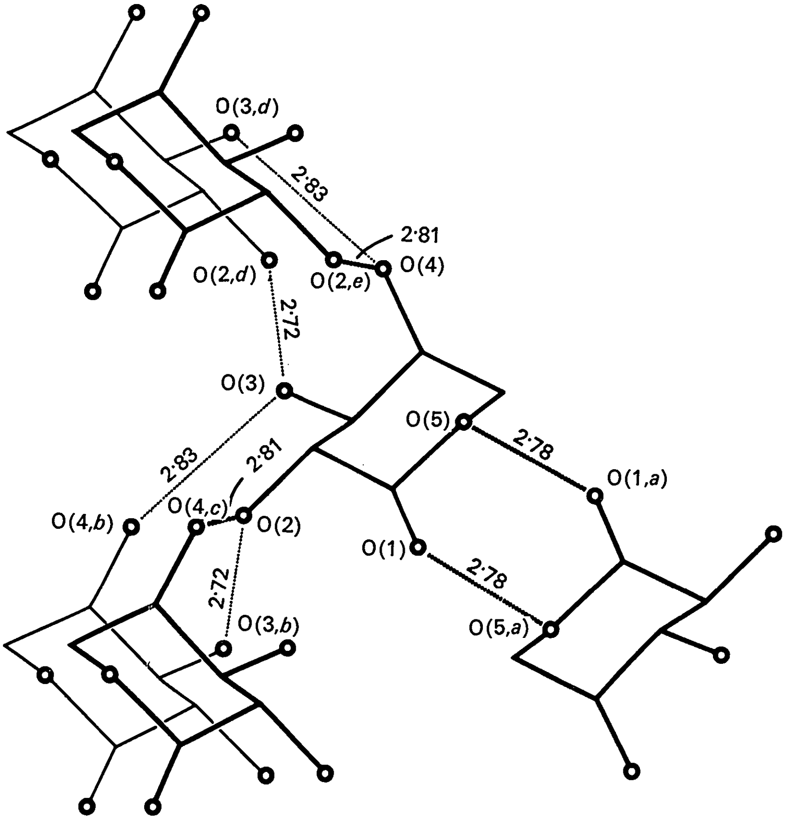

Fig. 3. Hydrogen bonding around one molecule. Dotted lines represent hydrogen bonds (See Table 7 for symmetry code).

Table 7. *Hydrogen bond distances and angles (with estimated standard deviations in parentheses)*

i	*j*	*k*	*d(ij)*	*d(jk)*	∠*(ijk)*
C(1)	O(1)	O(5,*a*)		2·779 (4) Å	103·9 (2)°
C(2)	O(2)	O(3,*b*)		2·724 (3)	110·9 (2)
C(2)	O(2)	O(4,*c*)		2·809 (3)	101·7 (2)
O(3,*b*)	O(2)	O(4,*c*)	2·724 (3) Å	2·809 (3)	130·1 (2)
C(3)	O(3)	O(4,*b*)		2·834 (3)	109·2 (2)
C(3)	O(3)	O(2,*d*)		2·724 (3)	116·4 (2)
O(4,*b*)	O(3)	O(2,*d*)	2·834 (3)	2·724 (3)	113·3 (2)
C(4)	O(4)	O(2,*e*)		2·809 (3)	120·8 (2)
C(4)	O(4)	O(3,*d*)		2·834 (3)	133·0 (2)
O(2,*e*)	O(4)	O(3,*d*)	2·809 (3)	2·834 (3)	101·1 (2)

Intermolecular non-bonded distances less than 3·3 Å between carbon and oxygen atoms

i	*j*	*d(ij)*
O(5)	O(2,*e*)	3·30 Å
O(1)	O(3,*b*)	3·16

Symmetry code

—	*x*	*y*	*z*
a	$-x$	$-y$	$1{\cdot}0-z$
b	$-1{\cdot}0-x$	$-0{\cdot}5+y$	$1{\cdot}5-z$
c	$-x$	$-0{\cdot}5+y$	$1{\cdot}5-z$
d	$-1{\cdot}0-x$	$0{\cdot}5+y$	$1{\cdot}5-z$
e	$-x$	$0{\cdot}5+y$	$1{\cdot}5-z$
f	$1{\cdot}0+x$	y	z

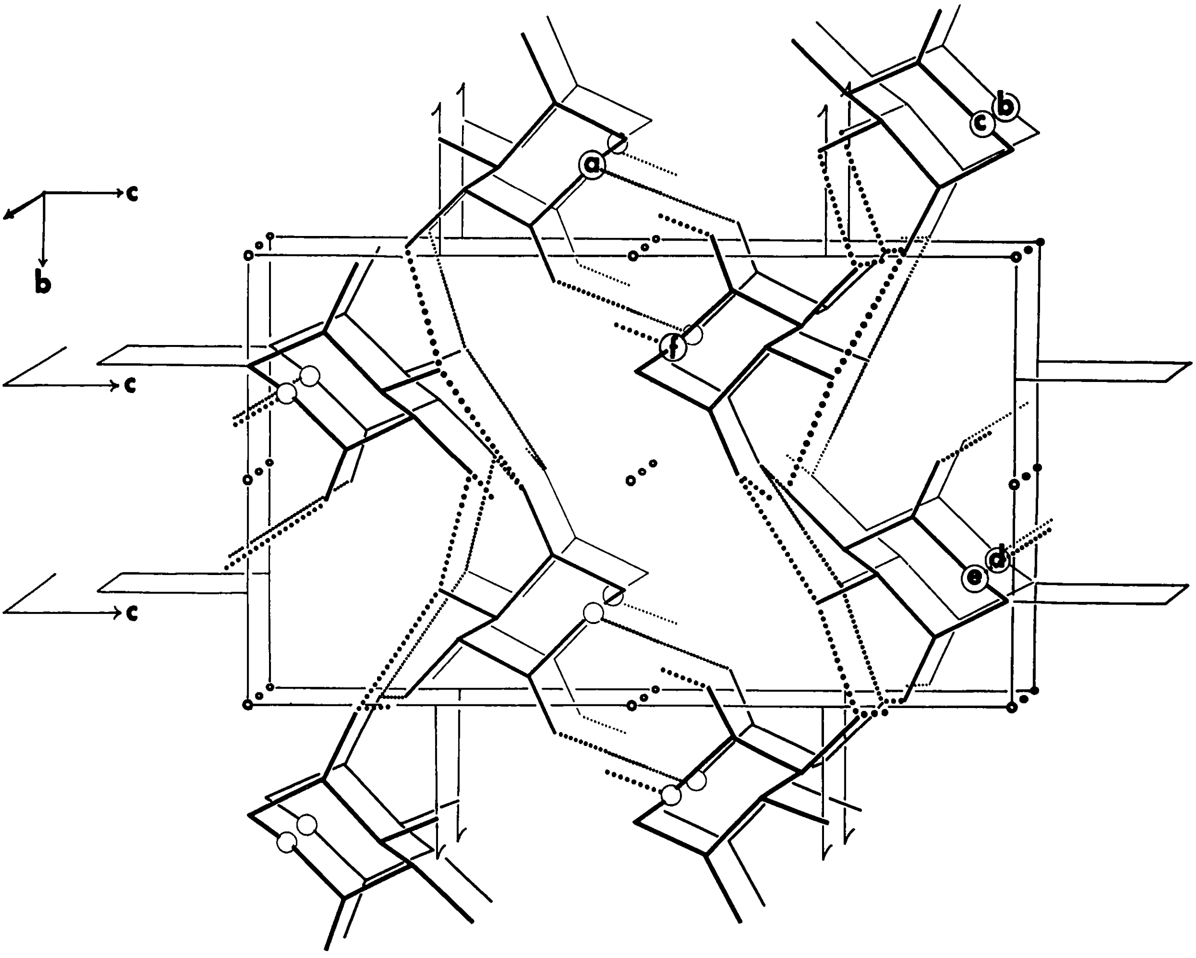

Fig. 4. Perspective drawing of the crystal structure (alphabetic symbols represent symmetry code given in Table 7).

The hydrogen bonding in the DL structure is extensive and involves all the oxygen atoms. The distances and angles are shown in Table 7. The left and right handed molecules are linked in pairs across centers of symmetry by a hydrogen bond from O(1) to O(5), the ring oxygen atom, as shown in Figs. 3 and 4. These DL pairs are then linked into a three-dimensional network by a donor/acceptor chain of hydrogen bonds which interconnect molecules of the same sense through the sequence

$$\rightarrow \mathrm{O}(2) \rightarrow \mathrm{O}(3, d) \rightarrow \mathrm{O}(4) \rightarrow \mathrm{O}(2, e) \rightarrow \mathrm{O}(3, f) \rightarrow \mathrm{O}(4, c) \rightarrow \mathrm{O}(2) \rightarrow .$$

These hydrogen bonded chains linking a sequence of D or of L molecules are therefore also centrosymmetrically related. There are two non-bonded O···O distances which are 3·16 and 3·30 Å and these are also shown in Table 7.

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The Structure of 1,3-Diamino-2,4,6-trinitrobenzene, Form I.

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The crystal structure of 1,3-diamino-2,4,6-trinitrobenzene, form I, has been determined by X-ray diffraction. The dimensions of the monoclinic unit cell are: $a_0 = 7{\cdot}30$, $b_0 = 5{\cdot}20$, $c_0 = 11{\cdot}63$ Å, $\beta = 95{\cdot}9°$. The space group is *Pc* with two molecules per cell. The molecule is approximately planar with apparent intramolecular hydrogen bonds between adjacent amino and nitro groups. The benzene ring is distorted so as to relieve the overcrowding of the planar configuration. The molecules are linked by intermolecular hydrogen bonds into continuous chains.

Introduction

Two crystalline polymorphs of 1,3-diamino-2,4,6-trinitrobenzene have been identified by X-ray powder diffraction. Form I is stable from room temperature to 217°C and form II from 217°C to its melting point, 286°C. The solid–solid transition is rapid on heating, and the transition temperature was determined by